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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

Å	angstrom	μm	micrometer
ft	foot	wt %	weight percent
min	minute		

THE PHASE RELATIONSHIP OF TALC AND AMPHIBOLES IN A FIBROUS TALC SAMPLE

By Robert L. Virta [1]

ABSTRACT

The Bureau of Mines examined a fibrous talc sample from the Gouverneur talc district in New York by transmission electron microscopy (TEM) and polarized light microscopy to determine the mineralogical relationship of the fibrous talc to the amphiboles present in the sample. Two amphiboles, anthophyllite and tremolite, were present in the sample. Tremolite occurred as a separate mineral phase, which was blocky in habit. Only a few composite tremolite-talc grains were observed. Anthophyllite, however, was present only in the fibrous talc grains. Microdiffraction study of the fibrous talc grains containing anthophyllite showed that the anthophyllite was intermixed with the talc on a fine scale and that there was a crystallographic relationship between the talc and anthophyllite lattices in the fibrous talc grains. A mechanism similar to the process that forms biopyriboles could explain the structural defects, the fibrous habit of the talc, and the structural relationship between the talc and anthophyllite in the fibrous talc grains. Because of these characteristics, phase contrast microscopy and a provisional TEM technique for monitoring asbestos exposure would not distinguish between fibrous talc and fibrous amphiboles. TEM techniques employing electron diffraction and energy-dispersive X-ray analysis are recommended to positively identify the fibrous phases for regulatory purposes.

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INTRODUCTION

Health scientists are interested in occurrences of amphiboles in talc deposits because of the apparent association between cancer risk and the inhalation of pure amphibole asbestos (21).[2] Among the lesser studied occurrences are the fibrous amphiboles associated with the fibrous talc in the Gouverneur talc district, St. Lawrence County, NY.

Fibrous amphiboles are present in minor amounts throughout deposits in the Gouverneur district. In some localities, they have been altered through metamorphism (5, 8, 19). It is these amphiboles that are associated with fibrous talc (5, 8). Two fibrous talc samples from St. Lawrence County, were found to contain composite talc-amphibole fibers, rather than pure talc. In one case, tremolite occurred with talc; in the other, an unidentified Mg amphibole was present. In both cases, a crystallographic relationship between the talc and amphibole lattices was observed (1, 22). Studies of a partially altered fibrous anthophyllite from Vermont have also shown that chain width disorder and intergrown sheet structures are sometimes found in the amphibole structure (23, 25). Missing structural units were explained as providing sites for ion migration and structural reordering to form the sheet silicate structures from the amphibole double-chain structure (24).

Asbestiform amphiboles, whether occurring with fibrous talc or not, are monitored because of the health risk they pose (4, 9, 14-15). Monitoring is performed using phase contrast microscopy; particles that are equal to or longer than 5 μm and that have a length-to-width ratio greater than 3 to 1 are classified as asbestos (18). When fibrous amphiboles occur with platy talcs, this monitoring process is relatively definitive for asbestos because of the morphological differences. It is when fibrous amphiboles occur with fibrous talc that morphology alone is inadequate to distinguish between phases (16). For this reason, TEM has been recommended for regulatory use. Particle morphology, electron diffraction (ED) and energy dispersive X-ray analysis (EDX) are used to positively identify the particles (6). To provide a relatively rapid analysis for regulatory use, a provisional Environmental Protection Agency (EPA) technique that relies on particle morphology, EDX, and qualitative ED for particle identification was developed (20). In this technique, a 5.3-Å repeat spacing parallel to the long axis of the fiber and an Mg and Si spectrum are used to classify a particle as the amphibole anthophyllite. These characteristics, however, are similar to those of fibrous talc and could result in the misidentification of talc as anthophyllite.

The purpose of this Bureau of Mines study is to determine the phase relationships between the talc and amphiboles present in the sample and examine possible problems that could be encountered in monitoring for asbestos because of the presence of fibrous talc.

ACKNOWLEDGMENTS

The assistance of Dr. A. G. Wylie, crystallography and mineralogy, and C. W. Huggins, X-ray diffraction, both of the Bureau of Mines Avondale Research Center is gratefully acknowledged.

EXPERIMENTAL WORK

SAMPLE AND SAMPLING REGION

The sample selected for this study is a coarsely ground fibrous talc sample from Talcville, St. Lawrence County, NY. The talc deposits in this area consist of lenses of talc interbedded with metasedimentary and metasomatic rocks of the

[2]Underlined numbers in parentheses refer to items in the list of references at the end of this report.

Precambrian Grenville series. Talc, tremolite, anthophyllite, serpentine, chlorite, mica, quartz, and diopside are present in deposits along the talc belt (2-3 5, 8, 13, 19). Most of the talc and the amphiboles formed through the prograde metamorphism of quartzite and dolomite. Anthophyllite and talc have also been reported to have formed through the retrograde alteration of tremolite (19). Engel (8) reported the occurrence of fibrous talc and serpentine which are pseudomorphous after tremolite as a late-stage reaction product of the dynamothermal metamorphism of the region. Stemple (22) examined one sample of talc from St Lawrence County by electron microscopy and reported the occurrence of fibrous talc-tremolite particles. They observed a crystallographic relationship between the fibrous talc and tremolite. Barr (1) reported the presence of amphibole-talc particles in a talc sample from St. Lawrence County. The amphibole was identified only as a Mg-rich monoclinic amphibole.

The sample used in this study was randomly selected. Consequently, it may not be representative of the morphology or the morphological characteristics of particles in the entire deposit.

SAMPLE PREPARATION

Samples were prepared for X-ray diffraction analysis (XRD) by freezer milling to minus 325 mesh and briquetting in a pellet press. Quartz present in the sample was used as an internal calibration standard.

Samples for infrared spectrophotometric analysis (IR) were prepared by freezer milling to minus 325 mesh and mixing 2 mg of sample in 200 mg of KBr powder in a mixer mill. Pellets were made in a vacuum press under 9 tons of pressure for 2 min.

Samples for TEM analysis were ground and suspended in water with sonification. A drop of the suspension was placed on a collodian-coated TEM grid, dried on a hotplate, and carbon-coated in a vacuum evaporator.

ANALYSIS AND RESULTS

Talc, quartz, tremolite, phlogopite, and carbonate were identified by polarized light microscopy (PLM). Talc is the major phase with 1 to 3 wt % quartz and 1 to 3 wt % tremolite present. Phologite and carbonate were trace constituents. Talc particles had lengths ranging from 5 to several hundred micrometers. The particle morphology of the talc ranged from platelets to individual fibers to fiber bundles with splayed ends (fig. 1). The

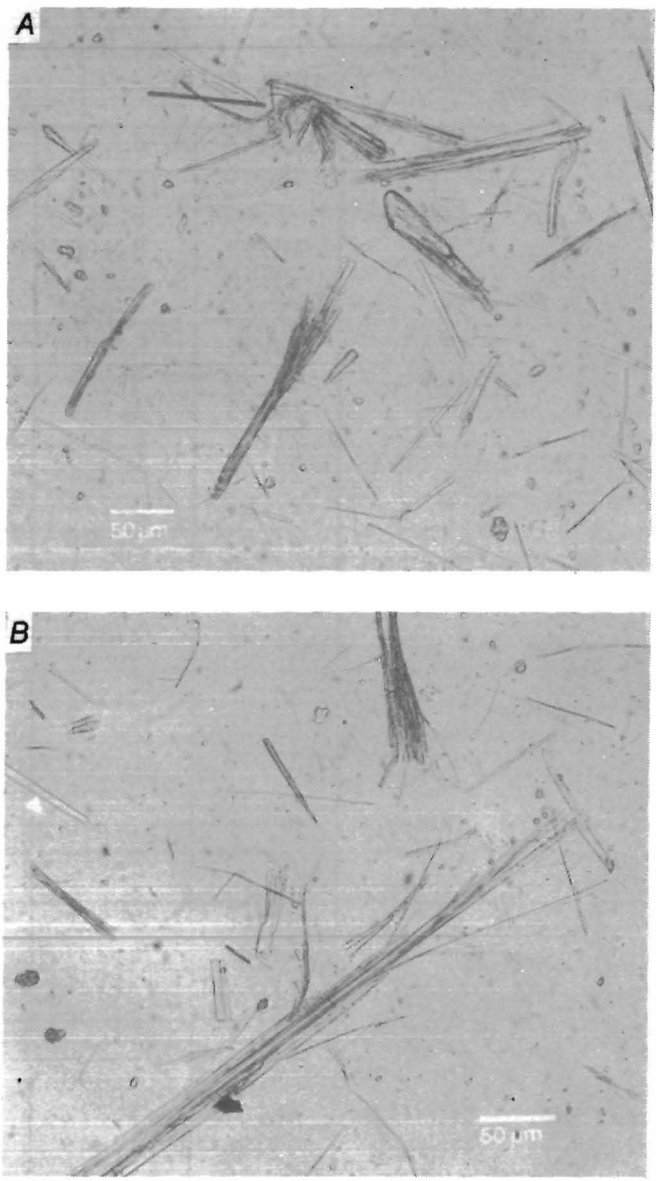

FIGURE 1. - *A*, Variation of particle sizes and shapes in the talc sample, including platelets and fiber bundles of talc; *B*, talc fiber bundles splaying into thin talc fibrils.

quartz and tremolite had particle sizes of 10 to 150 μm. Quartz grains were generally anhedral and often contained inclusions of fibers. The tremolite was present in a blocky habit with a few prismatic grains. Several tremolite particles had partially altered to talc, forming composite talc-tremolite grains similar to those described by Wright (27). No gradations in the refractive indices between minerals were observed in these composite grains or in the fibrous talc particles. Gradations in the refractive indices have been reported in particles where amphibole are altering to talc (1).

Talc, quartz, tremolite, anthophyllite, and phlogopite were identified using XRD (table 1). Slightly less than 5 wt % anthophyllite was estimated to be present using mixed standards. Tremolite, whose major peak intensities were less than those of anthophyllite, was present in lesser concentration than anthophyllite. The maximum concentration of 5 wt % for anthophyllite was verified using mixed standards for IR analyses.

TABLE 1. - Powder X-ray diffraction data, briquetted fibrous talc

hkl	2θ[1]	d_{obs}	d_{111}[2]	I	Other minerals
	8.83	10.014		45	Phlogopite.
002	9.44	9.368	9.34	>100	Talc.
	10.53	8.400		20	Tremolite.
	10.70	8.267		8	Anthophyllite.
	17.66	5.021		6	Phlogopite.
004	18.96	4.680	4.66	85	Talc.
020	19.40	4.575	4.55	20	Talc.
	20.85	4.260		25	Quartz.
	21.11	4.208		5	Tremolite.
	26.66	3.344		>100	Quartz.
	27.23	3.274		6	Tremolite
	27.65	3.226		12	Anthophyllite.
006	28.60	3.121	3.116	>>100	Talc.
	29.33	3.044		64	Anthophyllite.
	31.92	2.803		8	Tremolite.
	32.64	2.743		7	Anthophyllite?
	35.30	2.542		3	Anthophyllite.
	35.76	2.510		4	Phlogopite.
132	36.18	2.482	2.476	3	Talc.
	36.55	2.458		20	Quartz.
008	38.46	2.340	2.335	11	Talc.
	38.83	2.319		3	Tremolite or anthophyllite.
	39.48	2.282		8	Quartz.
	40.31	2.237		6	Quartz.
	42.47	2.128		8	Quartz.
	45.14	2.008		14	Phlogopite.
	45.81	1.980		7	Quartz.
	48.13	1.890		18	Tremolite.
0•0•10	48.61	1.872	1.870	35	Talc.
	49.38	1.845		6	Anthophyllite?
	50.20	1.817		20	Quartz.
	54.91	1.672		11	Quartz.
313	58.20	1.585		22	Talc?

See footnotes at end of table.

TABLE 1. - Powder X-ray diffraction data, briquetted fibrous talc--Continued

hkl	2θ[1]	d_{obs}	d_{lit}[2]	I	Other minerals
0•0•12	59.20	1.560	1.557	16	Talc.
	60.03	1.541		24	Quartz.
060, $33\overline{2}$	60.68	1.526	1.527	17	Talc.
062, 330	61.68	1.503	1.509	6	Talc.
	64.88	1.437		2	Tremolite.
	67.80	1.382		15	Quartz.
	68.20	1.375		25	Quartz.
	68.36	1.372		19	Quartz.
	68.50	1.369		4	Unidentified.
	69.38	1.354		11	Talc?
0•0•14	70.33	1.338	1.336	14	Talc.
260	71.43	1.320	1.317	4	Talc.
	73.53	1.288		4	Quartz.
	75.73	1.256		7	Quartz.
	77.73	1.228		4	Quartz.
	79.96	1.199		5	Quartz.
	80.75	1.190		4	Unidentified.
	81.13	1.183		5	Quartz.
	81.53	1.180		8	Quartz.
	83.88	1.153		3	Quartz.

[1]Naturally occurring quartz found in talc fibers used as internal standard for minor 2-theta corrections.
[2]Values from JCPDS card 13-558.

Based on the results of the PLM, XRD, and IR analyses, the sample is estimated to be composed of greater than 90 wt % talc, 3 to 5 wt % anthophyllite, 1 to 3 wt % tremolite, 1 to 3 wt % quartz, and trace amount of phlogopite and a carbonate.

Although tremolite and anthophyllite were detected by XRD, only tremolite was observed by PLM. This suggests that anthophyllite is closely associated with the talc on a submicroscopic scale. Electron microscopy (EM) further confirmed this relationship of the anthophyllite to the talc. The morphological characteristics observed by PLM were observed by TEM on particles whose size was below the resolution limit of PLM (fig. 2). Both platy and fibrous particles were observed. Several fiber bundles composed of fine fibrils were also present. The individual fibrils are approximately 1,000 Å wide.

Identification of the individual fibers was performed using selected area electron diffraction (SAED) and EDX. The SAED spot patterns for various particle orientations of talc, tremolite, and anthophyllite were plotted using the reflection conditions specified in the

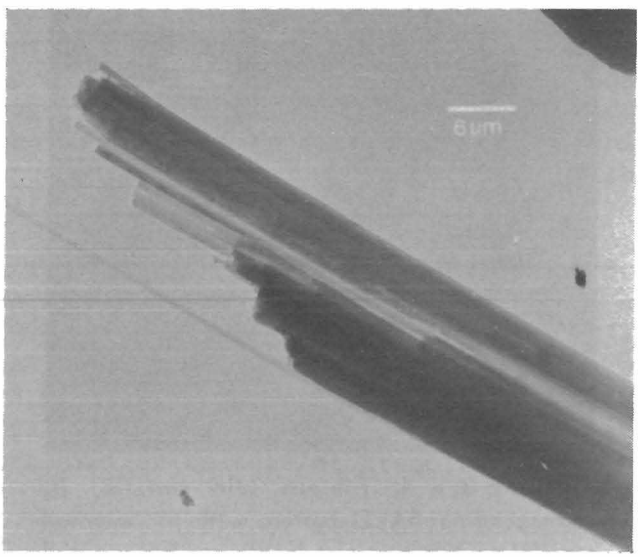

FIGURE 2. - Fibrous talc particle pseudomorphic after anthophyllite. Note the narrow fibrils of talc separating from the particle.

International Tables for X-ray Crystallography (10). These predicted SAED patterns and calculated d spacings were then used to identify the mineral phases. For tremolite, the body-centered cell described by Warren (26) was used to determine reflection conditions.

All fibrous particles were identified as either talc or talc-amphibole particle using SAED and EDX. Figures 3 and 4 show an elongated talc particle and a fibrous talc-amphibole particle respectively. The talc is oriented with its b*

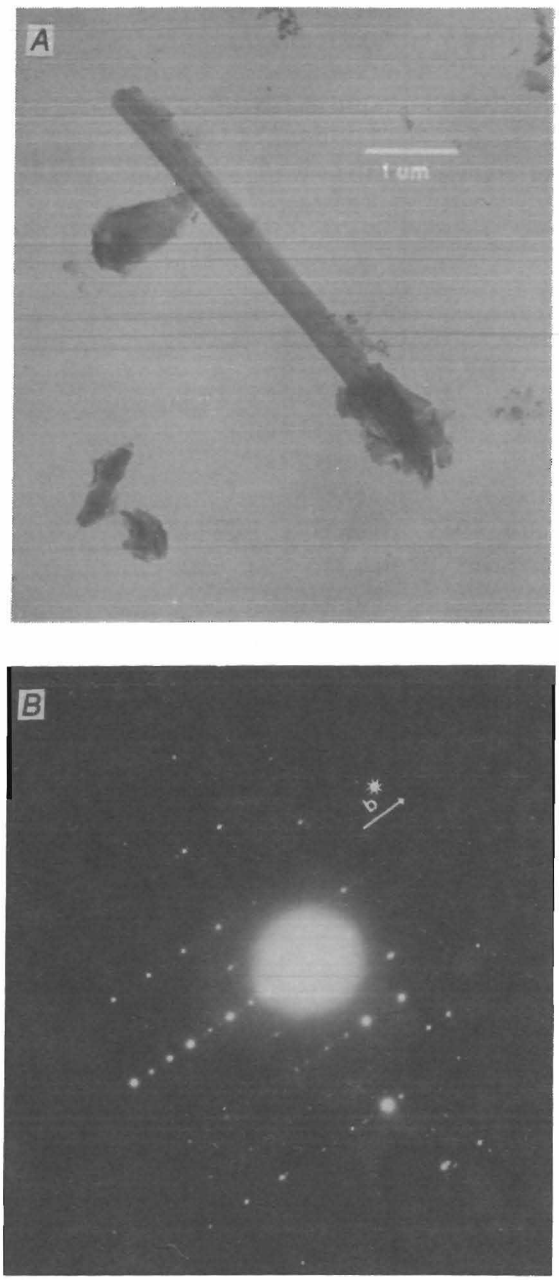

FIGURE 3. - *A*, Fibrous talc particle; *B*, corresponding SAED pattern with a* oriented parallel to the particle length and b* oriented perpendicular to the particle length (TEM photomicrographic).

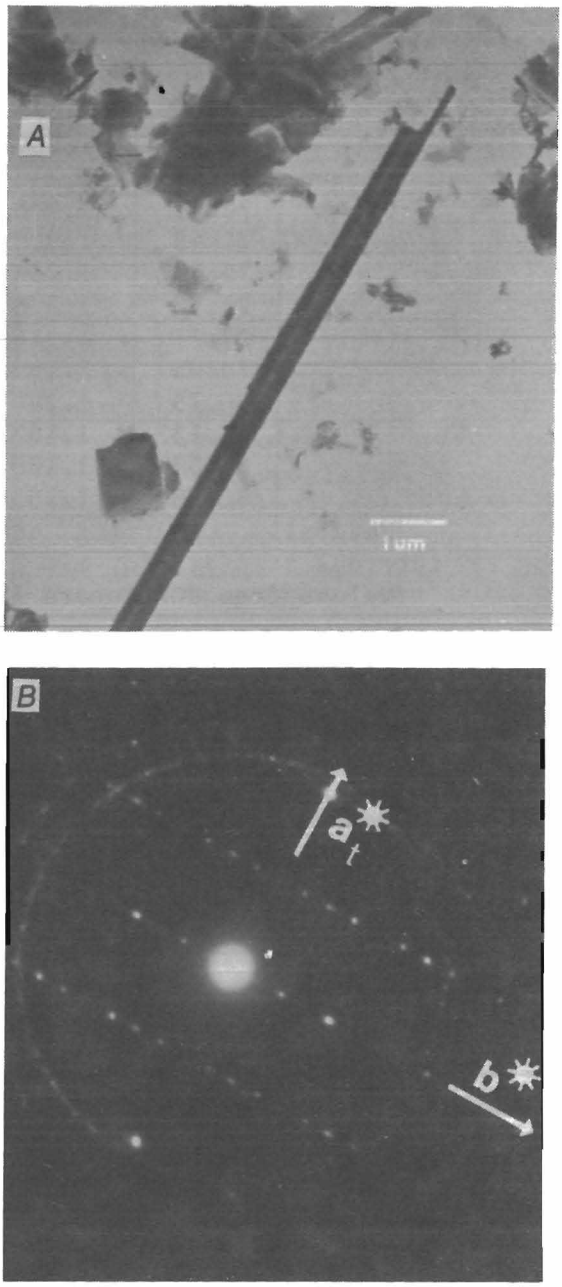

FIGURE 4. - *A*, Fibrous talc-amphibole particle; *B*, corresponding SAED pattern with a* (talc) and c* (amphibole) parallel to the particle length and b* (talc) and b* (amphibole) perpendicular to the particle length (TEM photomicrographic).

direction perpendicular to the length of the fiber and its a* direction parallel to the fiber length (fig. 3*B*). For talc-amphibole particles, the amphibole ED pattern is superimposed on the talc pattern (fig. 4*B*). The amphibole is oriented with the c* direction parallel to the fiber length and b* perpendicular to the fiber length. This ED pattern is shown schematically in figure 5, where the b* talc axis is parallel to the b* amphibole axis and the a* talc axis is parallel to the c* amphibole axis. The a* amphibole axis and c* talc axis were parallel to the electron beam. Dominant orientation of the (100) amphibole face perpendicular to the electron beam has been reported in the literature for amphibole asbestos (17). The maximum crystal growth of the fibrous talc is along the a axis with limited growth along the b axis and c axis. In platy talcs, the a and b lattice directions usually have equivalent crystal growth (7).

Many of the fibrous talc-amphibole particles exhibit a streaking of the ED pattern in the b* (amphibole) direction, suggesting possible defect structures in the b* lattice direction (fig. 4*B*). Other fibrous particles displayed distinct ED spot patterns, indicating that structural defects were minimal. These generally exhibited only a talc ED pattern.

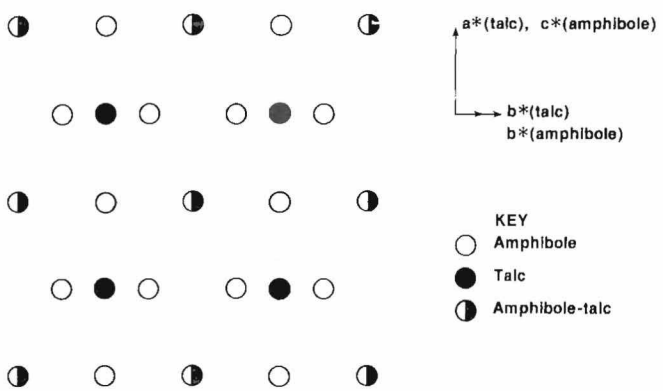

FIGURE 5. - Representation of superimposed talc-amphibole reciprocal lattices show in figure 4*B*. a* (talc) is parallel to c* (amphibole), and b* (talc) is parallel to b* (amphibole). b* (talc) ≈1/2 b* (amphibole).

Microdiffraction (μD) was used to determine if the mixed talc-amphibole ED pattern observed using conventional SAED could be attributed to alteration on a submicroscopic scale or partial alteration of an entire portion of the individual fibrous grains. Microdiffraction images were observed using a nominally rated 200-Å-diameter electron beam in the scanning transmission electron microscope mode along both the length and the width of the particles. Most of the fibrous particles exhibited either a talc μD pattern or a mixed talc-amphibole μD pattern. Only a few fibrous particles exhibited a mixed talc-amphibole μD pattern in one portion of a grain and a talc μD pattern in another portion of the same grain. No fibrous particles exhibited only an amphibole μD pattern.

Approximately 50 fibrous particles were examined qualitatively using EDX and found to be composed primarily of Mg and Si. Less than 2% of the particles contained any Ca or Fe, suggesting that tremolite is a minor to trace phase and that anthophyllite is the predominant amphibole. Semiquantitative EDX analyses were performed on particles with thicknesses less than approximately 1 μm using the Cliff-Lorimer technique (11). A pure Italian talc sample was used as the EDX standard. The Italian talc was composed of 19.8 wt % Mg and 29.0 wt % Si as determined using wet chemical techniques. Accuracy of the Cliff-Lorimer technique was approximately ±5 wt % relative under the operating conditions used in this study. A 4.5 wt % water content of the talc was assumed.

The fibrous particles ranged from 18.2 to 21.2 wt % Mg with an average value of 20.4±0.6 wt %. Silicon ranged from 27.9 to 30.6 wt % with an average value of 28.9±0.6 wt %. No Fe was detected. Several blocky particles exhibited EDX spectra similar to that of tremolite.

The percentage of amphibole in each fibrous particle could not be determined accurately using EDX because of the similarity of the Mg and Si content of the

talc and amphiboles. The particles would have to contain approximately 40 wt % anthophyllite before any significant shift in talc composition would be observed using semiquantitative techniques. The presence of more than 5 wt % tremolite would be detected because of the presence of Ca in the EDX spectrum. No significant variations in elemental concentrations (less than 2 wt %) were detected in different portions of most analyzed grains.

DISCUSSION

The intergrowth of talc with amphiboles, serpentine, chlorite, mica, and pyroxenes has been reported in several papers (1, 22-23, 25). The crystallographic relationship between the amphibole and fibrous talc in the sample examined is similar to that observed by Stemple (22), except that anthophyllite and possibly tremolite is intergrown with talc rather than only tremolite. Since both tremolite and anthophyllite are found in talc deposits and retrograde alteration of amphiboles has been reported in geological studies of the sampling area, the alteration of either mineral to fibrous talc could be expected.

Within the fibrous portion of the sample, the EDX and ED data indicate that no amphiboles occur as a free phase and that amphiboles occur only as composite talc-amphibole grains. The amphibole structures were determined to be interspersed on an extremely fine scale (several hundred angstroms in width) using μD and EDX. In all cases, either talc or superimposed talc and amphibole diffraction patterns were observed. No diffraction patterns and EDX spectra corresponding to a pure amphibole phase were observed. Had the composite particles been composed of coarse lamallae of talc and amphibole or had partial alteration of an entire portion of a grain occurred, only ED patterns and EDX spectra typical of an amphibole would have been observed.

Veblen (25) describes structural defects in pyroxenes and amphiboles which are believed to contribute to the alteration of their chain structures. The missing structural units were explained as providing sites for ion migration and structural reordering in the amphibole to form the sheet structure (24). Similar structural changes are likely to have been involved in the alteration of the amphibole to fibrous talc. The crystallographic relationship between the fibrous talc and amphibole and the fine intermixing of the two minerals are consistent with such an alteration mechanism. Defect structures in the lattice structure parallel to the b* direction of amphibole suggested by the streaking observed in the ED patterns are also consistent with the chain width disorder of chain silicate alteration described by Veblen (25).

A positive identification of the amphibole within the talc fibers could not be made by EM because all fibers had their a axis of amphibole parallel to the electron beam and the a* spacing was not determined. Tilting of the sample through angles of ±25° had almost no effect on the ED pattern. This phenomenon is explained as a result of the ED formation process related to the particle thickness (12). However, the data indicate that anthophyllite, rather than tremolite, is the major amphibole occurring in the fibrous talc grains. The 1 to 3 wt % nonfibrous tremolite observed as a free amphibole phase by PLM represents the bulk of the tremolite in the sample. Intermixing of the anthophyllite with talc fibers could account for its not being observed as a free amphibole phase by PLM despite its presence in the sample in greater quantities than tremolite. This also suggests that the fibrous talc and fibrous talc-anthophyllite particles formed from anthophyllite. Further, the fibril dimensions and dominant (100) orientation of the amphibole lattice, similar to what would be observed with asbestos, suggest that the fibrous morphology is due to the alteration of fibrous, if not asbestiform, anthophyllite. The dominant (001) crystal face of the talc would be developed in the alteration products due to the nature of the alteration process.

Based upon the results of this study and of studies on fibrous talc from other regions of the Gouverneur talc district, fibers of talc or talc-amphibole composites could be expected as a result of the alteration of fibrous amphiboles. This is one area of consideration when regulating for asbestos in talcs. The current phase contrast technique for asbestos monitoring uses the criteria of length equal to or greater than 5 μm, aspect ratio equal to or greater than 3 to 1, and parallel sides for classifying particles as asbestos. Many of the fibrous talc particles observed would meet these criteria, so other means of evaluating air monitoring filters would be required in these cases. Individual fibers could be positively identified with TEM using particle morphology, ED, and EDX. The use of ED, however, requires photographing and indexing each ED pattern. For regulatory purposes, a more rapid TEM technique would be required to permit analysis of large numbers of samples. A proposed technique using ED and the 5.3-Å repeat spacing would be appropriate if fibrous talc was not present. The 5.3-Å c spacing is very similar to the 5.28-Å a spacing observed on the fibrous talc grains, and both are oriented parallel to the fiber length. The 5.28-Å spacing would be indistinguishable from the 5.3-Å spacing for amphiboles using qualitative techniques. The particle morphology and the Mg and Si composition are similar to those of fibrous anthophyllite. To increase the accuracy of this technique, the spacings perpendicular to the 5.3-Å spacing should be determined. Assuming that most fibrous talc grains would lie on the 00ℓ face as they were in this study, the 18-Å b spacing of amphiboles would be distinguishable from the 9-Å b spacing in talc. On grains without this orientation, the more thorough quantitative ED technique would be required. An accurate assessment of whether fibrous talc should be suspected to be present in the sample prior to TEM analysis could be accomplished by determining the mineralogy through PLM and XRD.

CONCLUSIONS

The fibrous talc sample contained talc, anthophyllite, tremolite, quartz, and carbonate. Anthophyllite is present only within the fibrous talc grains. The intermixing of the talc and anthophyllite on a submicroscopic scale and the crystallographic relationship between the talc and anthophyllite crystal lattices suggest an alteration mechanism similar to that observed in the formation of some biopyriboles. Tremolite, however, was observed as a physically distinct nonfibrous amphibole phase, and little, if any, was present within the fibrous talc grains. The fibrous morphology, the lattice relationships in the talc-anthophyllite intergrowths, and the dominant (100) orientation of the amphibole lattice are suggested to result from alteration of fibrous or asbestiform anthophyllite. The possible presence of fibrous talc in samples containing fibrous amphiboles suggests that phase contrast microscopy and qualitative TEM techniques alone may be inappropriate for the regulation of amphibole asbestos in talcs. These techniques would not positively distinguish between amphiboles and fibrous talc because of the characteristics of the fibrous talc resulting from the alteration of fibrous amphiboles.

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